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National Archives and Records Service  
Washington, D.C. 20408



January 29, 1970

STATINTL

[Redacted]  
Records Administration Officer  
Central Intelligence Agency  
Washington, D.C. 20505

STATINTL

[Redacted]  
Attached is National Bureau of Standards Report #10 152  
which is a brief summary covering the work of the past  
calendar year, the work in progress and plans for this year.

If there are any questions or suggestions regarding the  
reports and the work, you may contact me on code 13,  
extension 36464.

Sincerely,

*James L. Gear*  
James L. Gear  
Preservation Officer

Attachment

# **NATIONAL BUREAU OF STANDARDS REPORT**

10 152

## **DEVELOPMENT OF SPECIFICATIONS FOR ARCHIVAL RECORD MATERIALS**

Annual Report  
to  
National Archives  
General Services Administration



**U.S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS**

## NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards<sup>1</sup> was established by an act of Congress March 3, 1901. Today, in addition to serving as the Nation's central measurement laboratory, the Bureau is a principal focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. To this end the Bureau conducts research and provides central national services in four broad program areas. These are: (1) basic measurements and standards, (2) materials measurements and standards, (3) technological measurements and standards, and (4) transfer of technology.

The Bureau comprises the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Center for Radiation Research, the Center for Computer Sciences and Technology, and the Office for Information Programs.

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Office of Standard Reference Data—Clearinghouse for Federal Scientific and Technical Information<sup>4</sup>—Office of Technical Information and Publications—Library—Office of Public Information—Office of International Relations.

<sup>1</sup> Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.  
<sup>2</sup> Located at Boulder, Colorado 80302.

# NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

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10 152

## DEVELOPMENT OF SPECIFICATIONS FOR ARCHIVAL RECORD MATERIALS

William K. Wilson  
Paper Evaluation Section  
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Institute for Applied Technology

Annual Report  
to  
National Archives  
General Services Administration

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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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## Development of Specifications for Archival Record Materials

### 1. BACKGROUND

In response to a request by the National Archives, the National Bureau of Standards is developing information on the variables associated with the stability of archival record materials, especially paper. Many record materials in repositories in the federal government and throughout the United States are in very bad physical condition. If proper specifications were available for materials that are designed to be used for permanent records, future problems would be greatly minimized and less resources would need to be devoted to repairing the mistakes of the past.

Although the present effort is directed toward paper, and especially the development of test methods that may be used in the preparation of specifications, other materials that will merit attention are quick copy reproductions from office copying machines, inks, typewriter ribbons, carbon paper, laminating film, mending tape and binding materials.

### 2. OBJECTIVES

The objectives of this program are as follows:  
(1) the development of information on the stability of paper and other record materials, (2) the development of test methods for the evaluation of the stability of paper and other record materials, (3) the development of specifications for these materials.

### 3. WORK IN PROGRESS

#### 3.1 Special Apparatus

In order to develop information on the relative stability of materials, it usually is necessary to devise a laboratory aging method that depends on a temperature higher than room temperature. The usual procedure is to use either ovens or oil baths. An oven is a very convenient aging device and it

allows the evaluation of large samples. However, temperature control in an oven usually leaves much to be desired, and it is not possible to maintain accurate control of relative humidity without the introduction of complicated auxiliary apparatus. For specification testing, an oven usually is considered to be satisfactory, but for research work an oven does not as a rule allow the proper temperature control.

For our purpose, it is considered necessary to maintain very accurate temperature and relative humidity control. Therefore, six new oil baths were constructed in the Bureau shop for maintaining constant temperature and relative humidity within very narrow limits. The temperature may be varied from ambient to about 100°C and the relative humidity may be varied from 0-100 percent. Although this type of apparatus requires considerable maintenance, it is very dependable and accurate.

Over the past two years, a complete facility for making handsheets has been installed. This apparatus also is used in work for another project. Special drying rings have been made so the experimental papers can be dried under proper restraint. The distilled water storage system has been enlarged to enable us to make especially pure papers in the laboratory. This will allow the addition of specific impurities to laboratory sheets in order to systematically study the effect of various materials that occur in paper from normal commercial manufacture.

### 3.2 Alum Sorption and Acidity of Paper

Acidity is the most important single variable associated with the permanence of paper. That which occurs in paper as a result of the manufacturing process is derived mostly from papermaker's alum, aluminum sulfate. The mechanism of the production of this acidity is not completely understood.

Considerable work has been done in this laboratory on the interaction of alum with a cellulosic ion exchange resin, which is used as a model compound in place of cellulose. The ion exchange resin contains a massive amount of a specific functional group, carboxyl, and this enables one to study the chemistry of these groups on a macro scale instead of resorting to micro analytical methods, as would be necessary with paper pulps containing smaller amounts of carboxyl.

From this work it has been learned that alum apparently produces acidity in paper by two different mechanisms. In the first mechanism, the aluminum only of the aluminum sulfate reacts with the carboxyl groups. When the ion exchange resin, which has been reacted with aluminum, is placed in water, it produces an acid reaction. If the resin is washed and again placed in water, it again produces an acid reaction. This can be repeated several times.

Acid also may be produced by another mechanism. One of the methods of adjusting acidity in the papermaking process is to add an alkali after alum has been added. This precipitates a complex aluminum compound, which includes sulfate, onto the fibers. This can produce sulfuric acid when the material is placed in water. The relative contributions of these two reactions to the acidity of paper and therefore to the degradation of paper is incompletely understood. Aging experiments will be carried out with paper which has been prepared to give these two types of reactions.

The analytical work in connection with the sorption of alum by papermaking fibers has been completed and a report is being prepared.

### 3.3 Thermal Analysis

NBS Report No. 10 113, Thermal Analysis of Modified Cellulose, has been distributed to the sponsors of this project. In this report, the techniques of differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) were used to study the effect of various chemical reagents that had been used to prepare modified celluloses. These modified celluloses are structures that could be found in papermaking pulps. As it is possible to obtain considerable information fairly quickly through the use of thermal methods, these methods are very useful for suggesting samples for further study by accelerated aging. Thermal analysis has been used as a screening test for developing information on probable stability. However, this will not supplant the technique of laboratory aging at lower temperatures, which does not destroy the paper.

DTA is a technique for studying the thermal behavior of materials as they undergo physical and chemical changes during heating. Differences in temperature between a cellulose or paper specimen and an inert material, as the two are heated at a constant rate, are plotted automatically on a chart. These temperature differences can mean many things depending on the nature of the sample. If energy is absorbed during the reaction, this part of the plot is called an endotherm; if energy is evolved, an exotherm. The shapes of the curves and the temperatures at which endotherms and exotherms occur may be interpreted in terms of stability. In this case, temperature differences were used to fingerprint the thermal decomposition of cellulose or chemical reactions that occur during decomposition.

TGA is a technique for measuring the weight loss of a material during heating. TGA data may give information on the relative thermal stability of cellulose modifications and on reactions that occur during heating.

It was found that the metal that reacts with the cellulose during paper manufacture has a very important bearing on the stability of the paper as measured by thermal analysis. Some of the cellulose modifications were less stable than others.

### 3.4 Laboratory Aging

All of the aging work has been done at 90°C and 50 percent relative humidity. It will be necessary, as the work progresses, to obtain data at other temperatures such as 80°, 70° and 60° and at other relative humidities. By proper selection of samples and by proper analysis of the data obtained at several temperatures, one perhaps can devise a simple laboratory aging method. This can be done only after obtaining the necessary data, which is a very time consuming process.

As it was considered necessary to concentrate on thermal analysis of modified cellulose and on the interaction of alum with papermaking pulps, no laboratory aging has been done during the past year. The work on thermal analysis has enabled the selection of samples for meaningful laboratory aging.

### 3.5 Hypochlorite Oxidation

Sodium or calcium hypochlorite is used extensively in the bleaching of papermaking fibers. The reaction of hypochlorite with cellulose is very complex and depends on a host of variables. Fortunately, considerable work has been published on this reaction and it has been possible to select a typical set of conditions for oxidation of cellulose with hypochlorite.

One or two high quality papermaking pulps will be selected and paper made from them. Portions of these pulps will first be reacted with hypochlorite to simulate an over-bleaching situation in which the cellulose is damaged. All of these papers then will be subjected to laboratory aging at various temperatures. This information should enable us to decide whether a simple laboratory aging method will be possible.

Although some work will be done with handsheets in connection with hypochlorite oxidation, the test conditions have been selected to enable us to have the work done in our semi-commercial paper mill, if this appears to be desirable.

### 3.6 Handsheets

The use of commercial papers in the evaluation of variables associated with the stability of paper always is open to question. It is almost impossible for a commercial mill to be able to supply all of the necessary information about the manufacture of any particular paper. Therefore, it is desirable, for research work, to make special papers in the laboratory. A commercial handsheet machine is available for use on this project.

It will be necessary to study the effect of various pressures during wet pressing on the properties of the paper. As it is likely that the bonding material in a paper degrades much more rapidly than the fibers, it is necessary to be able to control the extent of bonding. It is not easy to do this in a paper mill on an experimental basis.

### 3.7 Special Methods of Evaluation

The most common method of evaluating changes during aging of paper is folding endurance. Other methods that have been used are bursting strength, tearing strength, change in reflectance, and changes in other physical properties. Changes in acidity also have been measured.

In spite of the voluminous work that has been done on the aging of paper, practically nothing has been done to indicate the actual changes that occur in a sheet of paper upon aging. For example, little is known about the relative contributions of the degradation of the bonding of fibers and the degradation of the fibers themselves.

Changes in the structure of bonding may be studied by two different methods. Both of these methods assume that a procedure has been used to degrade the bonding after aging. This may be accomplished in a special flexing device that has been developed in the Paper Evaluation Section. If the bonding is degraded during the aging process, then flexing should destroy the bonding very quickly. The destruction of the bonding with flexing may be measured by (1) a change in the velocity of sound through the paper and (2) a change in the accessible surface area of the fibers. An apparatus for the determination of the velocity of sound (sonic modulus) through paper has been obtained. An apparatus for measuring the relative surface area (krypton sorption) is on order and should be available in about three months.

Degradation of the fibers may be determined in two ways: (1) single fiber strength determinations after defiberizing the paper and (2) zero span tensile strength of the paper. The measurement of single fiber strength is not at all easy and probably will not be done.

### 3.8 Manifold Paper

NBS Report No. 9959, Evaluation of the Stability of Manifold Papers, was distributed December 1968. In this report it was suggested that the specifications for manifold paper could be changed in order to ensure the procurement of more stable manifold papers. These recommendations involve setting the pH values higher than normally specified.

In order to obtain information on the physical properties of representative manifold papers, data will be obtained on the physical properties of several manifold papers. Some papers are on order and others will be ordered shortly. After these papers are tested, recommendations will be made for specifications for manifold papers for permanent records.

In order to determine the condition of manifold papers in file, the physical properties of several manifold papers, starting about 1924 when the Congressional Joint Committee on Printing first issued specifications for manifold papers, are being examined. It is hoped that this will allow a determination of the degradation that has occurred in manifold papers in file over the past 45 years. If meaningful data are developed from this survey, a report should be available by June 30.

#### 4. VISITS TO EUROPEAN LABORATORIES AND ARCHIVAL INSTITUTIONS

In connection with a trip to Oslo, Norway to a meeting of ISO/TC 6, Paper, SC 5, Testing Methods and Quality Specifications for Pulp, several visits were made to representative European archives and laboratories. This included visits to the Norwegian National Archives in Oslo, the Danish National Archives in Copenhagen, the German National Archives in Koblenz, Vezelinstituut T.N.O., Delft, Holland and the British Museum.

The workmanship in these various restoration laboratories is excellent. The stability of some of the materials used in restoration work may be open to question, but they are well aware of this.

Some excellent work is in progress at the British Museum. In addition to the "soluble nylon" method that has been developed to reinforce paper, they have developed methods for the cleaning and bleaching of paper.

Considerable research work on the preservation of records is in progress in countries behind the iron curtain. It should be useful to track down references to any of this work that has been published.

## 5. PLANS FOR CALENDAR YEAR 1970

Most of the plans for the coming year have been mentioned above. It is expected that the following will be accomplished during the year:

1. The report on alum sorption and acidity of paper should be available in February or March.
2. It is anticipated that thermal analysis will be used as an adjunct to the work on laboratory aging of various experimental papers. It is not likely that a special report on thermal analysis of these experimental papers will be issued. The data will be made available in any reports that may be issued on the aging of experimental papers.
3. The laboratory aging of several experimental papers will proceed during the year. A report should be available late in the year.
4. The work on manifold papers should be completed and a report should be available by June 30.
5. The feasibility of the special methods of evaluation mentioned above, namely sonic modulus and surface area, will be explored during the year. It is not likely that a study of these special methods will result in a separate report. These data probably will be included in reports on the aging of experimental papers.

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